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NEW OPTICAL METHOD FOR STUDYING DEFECTS IN SILICON

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A thesis submitted to Rensselaer Polytechnic Institute, Troy, New York, in partial fulfillment of the requirements for the degree of Master of Engineering.

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NEW OPTICAL METHOD FOR STUDYING
DEFECTS IN SILICON

by

Michael T. Mitchell

A Thesis Submitted to the Graduate
Faculty of Rensselaer Polytechnic Institute
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF ENGINEERING

Approved:

John C. Corelli

John C. Corelli, Thesis Advisor

Rensselaer Polytechnic Institute
Troy, New York

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ABSTRACT

A new optical method incorporating two monochromatic light beams incident on the sample whose temperature is at $\sim 78^{\circ}\text{K}$ is used to study infrared active radiation-induced defect absorption bands. Defect energy levels, photoionization excitation and de-excitation processes were examined for the divacancy associated bands found in the 3-3.6 μm wavelength region and two higher order bands which appear in the 7 to 14 μm wavelength region.

For the divacancy band region 10 Ω -cm and 0.1 Ω -cm n-type (P-doped) crucible grown silicon irradiated to 5×10^{18} neutrons/cm² ($E > 1$ MeV) were examined. An excitation energy (E_g) of width 0.81eV to 0.89eV was found to produce an absorption throughout the entire 3.3 μm absorption band region with a maximum absorption occurring around 3.1 μm . A model is proposed which incorporates direct band gap transitions.

A 200 Ω -cm p-type (B-doped) crucible grown silicon sample irradiated to 5×10^{18} neutrons/cm² ($E > 1$ MeV) and annealed at 500°C for 15 minutes was examined for higher order bands. The 9.54 μm and 9.08 μm bands were found to have a maximum absorption for band gap energy. A model is proposed for the photoionization process incorporating the band gap energy for absorption and a depletion energy of 0.74eV.

INTRODUCTION

A. Historical Background

One of the greatest contributions to modern technology has been the advent of solid state devices. Their use in almost every phase of electronics has revolutionized man's capability to understand and control his environment. Continuing research in the area of solid state will aid scientists and engineers in developing better, smaller, and more accurate electronic instrumentation needed to expand in all areas of science and industry. This continuing research will by necessity mean that better understanding of the solid state materials, their basic characteristics and physical behavior, under varied environmental situations will come about.

One semiconductor material in which property investigation is currently progressing is silicon, since it is one of the mainstays of solid state electronics. In particular, its behavior and changing characteristics in an environment of radiation has been under study due to its use in such capacities as reactor control, electronic control devices, radiation characterization, aerospace activities and other areas where a radiation environment exists. This radiation produces defects in the lattice structure, such as, vacancies, interstitials, line defects and many others which can alter the characteristics of the material by changing its conductivity, strength, ductility, etc. Continuing examination of these defects will give a better

understanding of how they come about, their influence on the material and how they may be promoted, eliminated or mitigated to serve man's needs.

A search of the literature indicates that since the time of Fan and Ramdas¹, much work has been performed on the study of defects by utilizing infrared spectroscopy. Infrared absorption bands have been studied and, together with other experimental techniques, such as, EPR, the bands have been linked to particular defects thereby aiding in understanding the defect's characteristics.

B. Statement of Problem

Some of these infrared absorption bands have been found to be electronic in nature and associated with transitions requiring an electron to be present in the ground state of the defect center. In particular those bands associated with the divacancy defect (Chen and Corelli)^{2,3} and the Higher Order Bands (Corelli, et. al.)⁴ have been found to have this characteristic. Previously when observing these bands, the ground state was populated by using the full energy spectrum (all wavelengths) of the light source and analyzing the absorption which occurred at a specific wavelength. This procedure did not allow one to determine the position of the energy level; therefore, the transition energy dependence remained unknown.

In this thesis, a new optical method for examining infrared absorption bands will be presented along with transition energy dependence results for the divacancy defect infrared absorption bands and the higher order bands.

II

EXPERIMENTAL METHOD

The new optical system introduced here eliminates the necessity of utilizing the full energy spectrum. Figure 1 depicts the experimental setup which employs two Model 98 Perkin Elmer monochromators. In contrast to previous experiments where the full energy spectrum strikes the sample and then the transmitted beam is examined by wavelength, only two wavelengths, with $\pm\Delta\lambda$ resolution, are incident on the sample. The first light beam (probe light) is incident normal to and passes through the sample on to a detector. The second beam (excitation light) strikes the sample slightly off normal incidence yet illuminating the same sample area as the probe light. With the probe light set at the predetermined wavelength of the absorption band to be studied, a pulse of excitation light whose energy can be varied (say from ~2.4eV down to 0.05eV) by the use of different prisms is shone on the sample. The variables available in the experiment are the excitation light energy, the pulse duration, the light intensity, the sample temperature and the polarization condition of probe and excitation light. The sample is kept in a cryostat for low temperature studies. Different types of cryostats with different windows, various types of detectors, prisms and sources can be utilized depending upon the specific experimental needs.

Using the optical system in Figure 1, results were obtained using a glass cryostat with NaCl windows and a copper cold finger

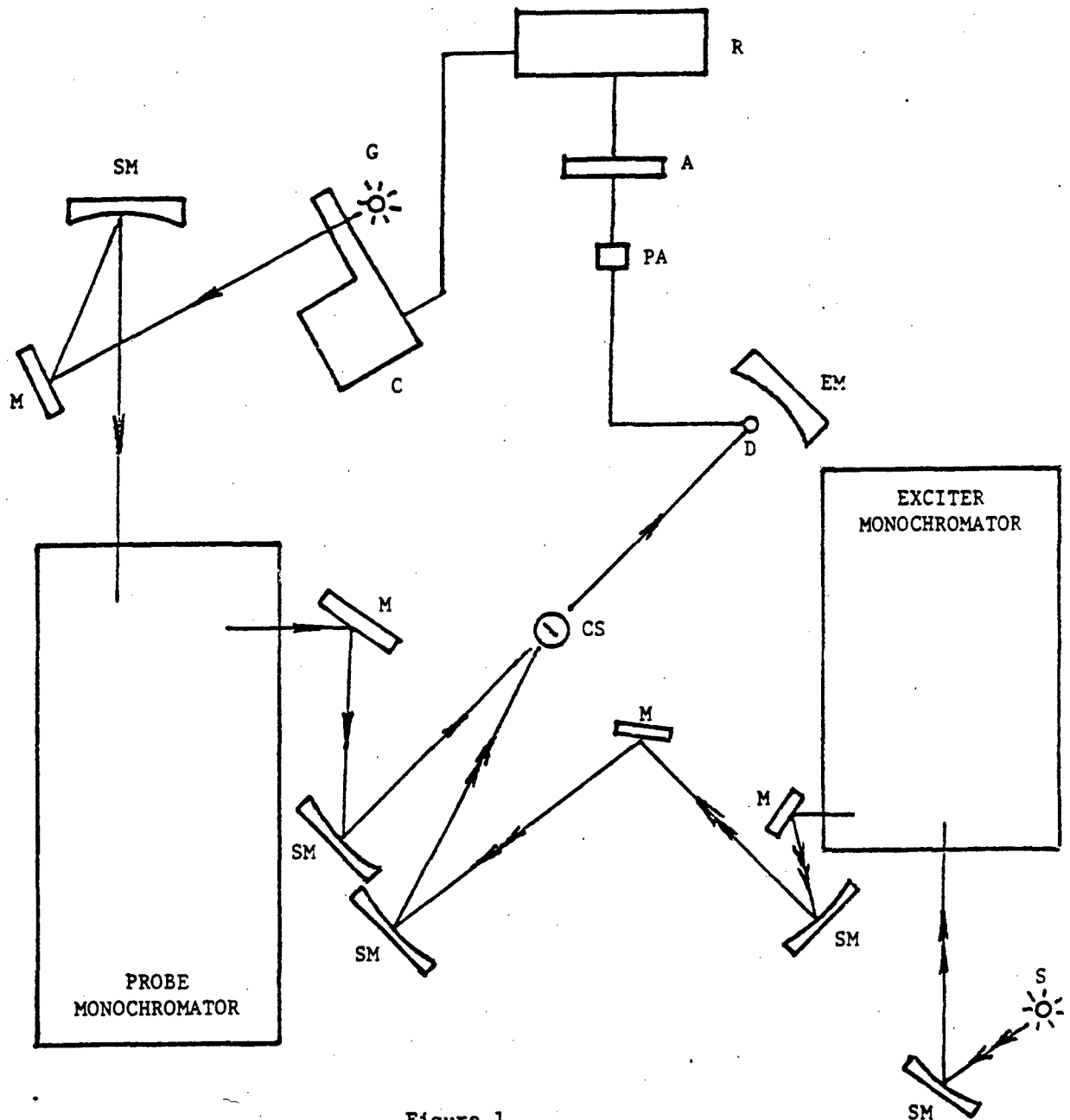


Figure 1

List of Instruments: p. 5

Figure 1 Instruments

- A - Amplifier
- C - Chopper at 13 cps
- CS - Glass Cryostat Holding Sample at 78°K
- D - Thermocouple Detector with KBr window
- EM - Ellipsoid Mirror
- G - Probe Light Source (Globar)
- M - Plane Mirror
- PA - Preamplifier
- R - Recorder
- S - Exciter Light Source (Sun Lamp with Quartz Envelope)
- SM - Spherical Mirror

to keep the sample at liquid Nitrogen temperature (78°K). A thermocouple detector with a KBr window was used for detecting the probe light and a globar was used for the probe light source. A tungsten lamp, globar, and high intensity quartz lamp were used alternately as sources for the excitation light. In addition, the optical system was made light tight by covering both monochromators and the path ways of both beams.

III

DIVACANCY BANDS

A. Experimental Results

The first infrared absorption band to be studied was the $3.62\mu\text{m}$ (2762 cm^{-1})² band associated with the divacancy defect.^{2,5} Two samples were utilized, each irradiated to 5×10^{18} neutrons/cm² ($E > 1\text{ MeV}$) of initial resistivity $0.1\Omega\text{-cm}$ and $10\Omega\text{-cm}$ n-type (P-doped) silicon crucible grown. Response of maximum absorption to the excitation light for the band was as depicted in Figure 2. There were at least two absorption and two decay modes noted for the effect of the excitation light; absorption $T_1 \sim 3.8\text{ sec}$; $T_2 \sim 12\text{ min}$; decay $T_3 \sim 50\text{ sec}$; $T_4 > 30\text{ min}$. Where $T_1 + T_2$ and $T_3 + T_4$ are the times to reach steady state. The energy range of the excitation beam giving the greatest absorption effect occurred from 0.81eV to 0.89eV . The experiment was conducted under light tight conditions, i.e., no light was allowed to strike the sample, both before and after the sample was brought to a temperature of 78°K . No other excitation energy was found to produce an absorption. It was also noted that the excitation energy was found to behave similar to a resonance and to have an effect as low as 0.56eV at the low energy tail and 0.95eV at the high energy tail.

The response of the $3.62\mu\text{m}$ probe energy to excitation energy is shown in Figure 3, in which the transmission of $3.62\mu\text{m}$ light through the sample is plotted as a function of the excitation energy. It was first believed that this excitation energy range was applicable to the $3.62\mu\text{m}$ band itself; however, it was found that the probe light alone

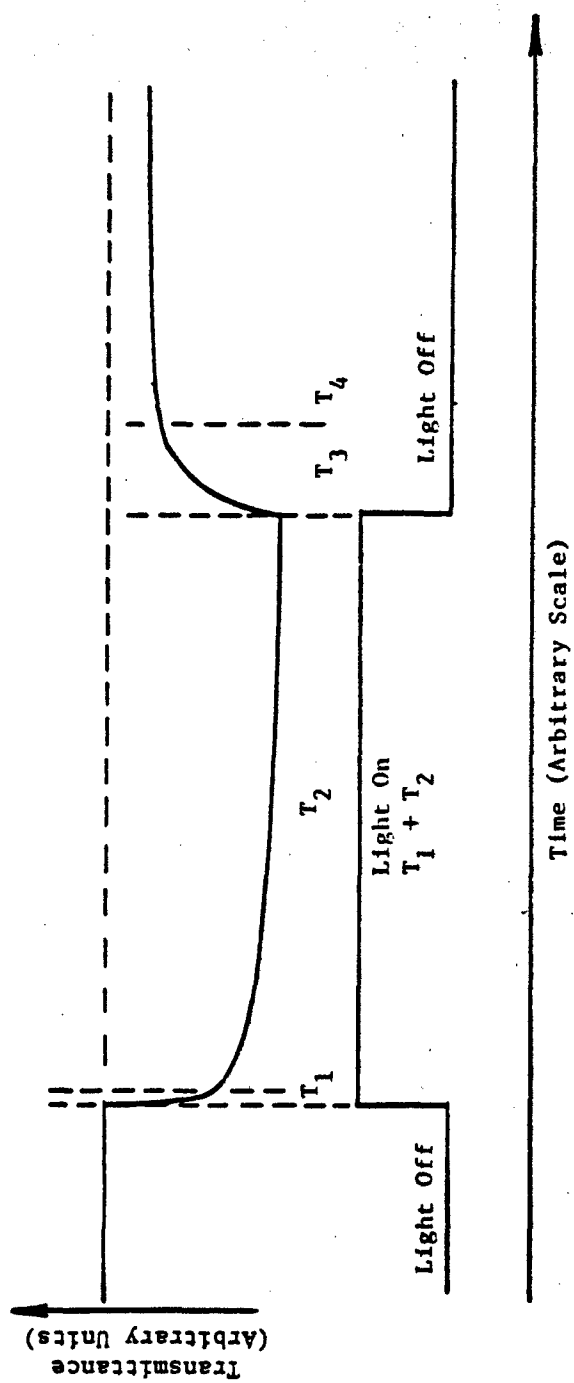


Figure 2.

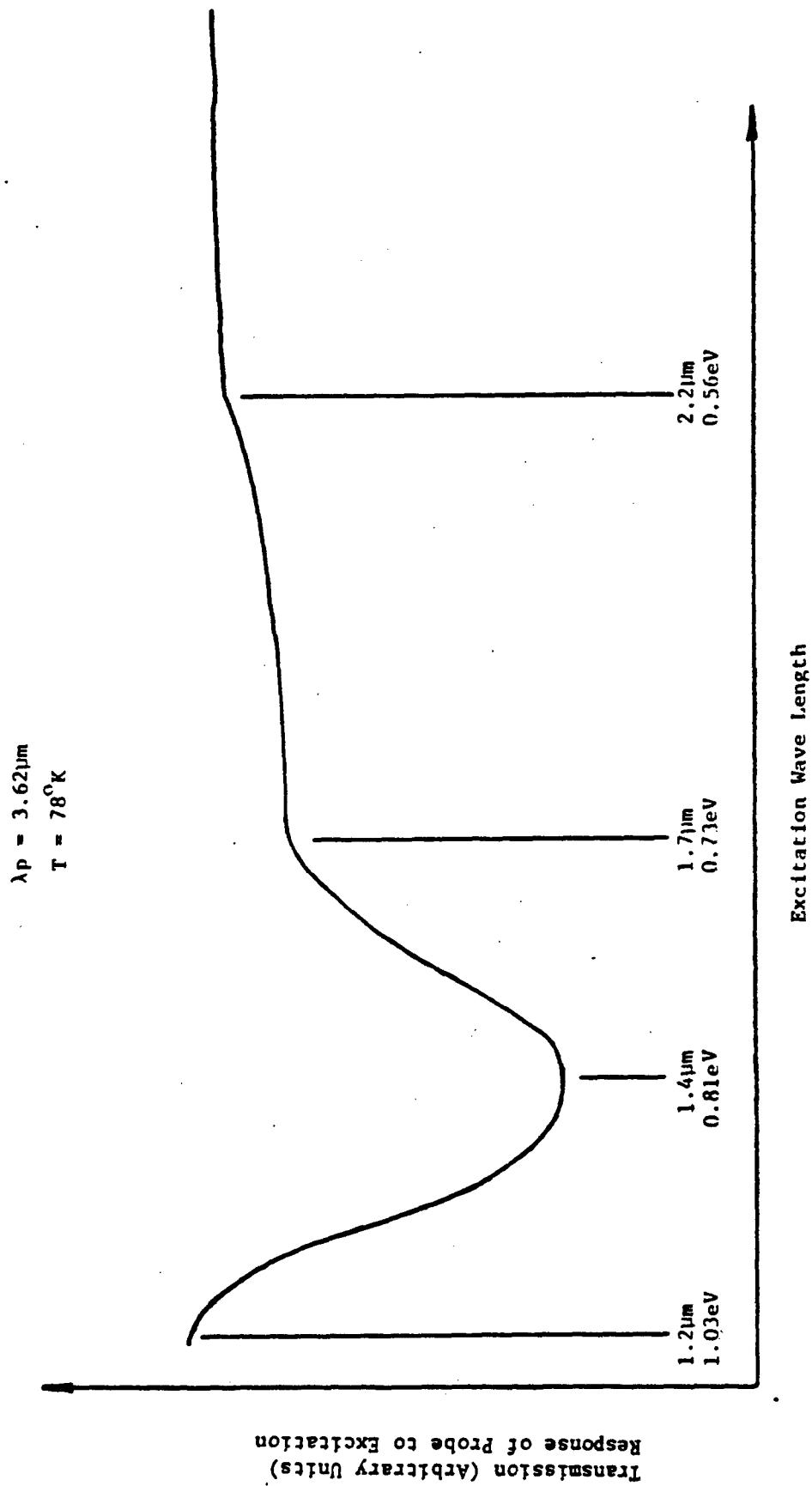


Figure 3

would weakly bring about the 3.62 μ m band and that the noted excitation energy affected not only the 3.62 μ m band but the entire 3.3 μ m region with a maximum absorption occurring around 3.1 μ m, see Figure 4.

Another experimental finding is that for the above samples near edge absorption occurred down to 0.6eV implying that the band gap (indirect optical) of Si has closed due to band tailing from 1.17eV down to 0.6eV.

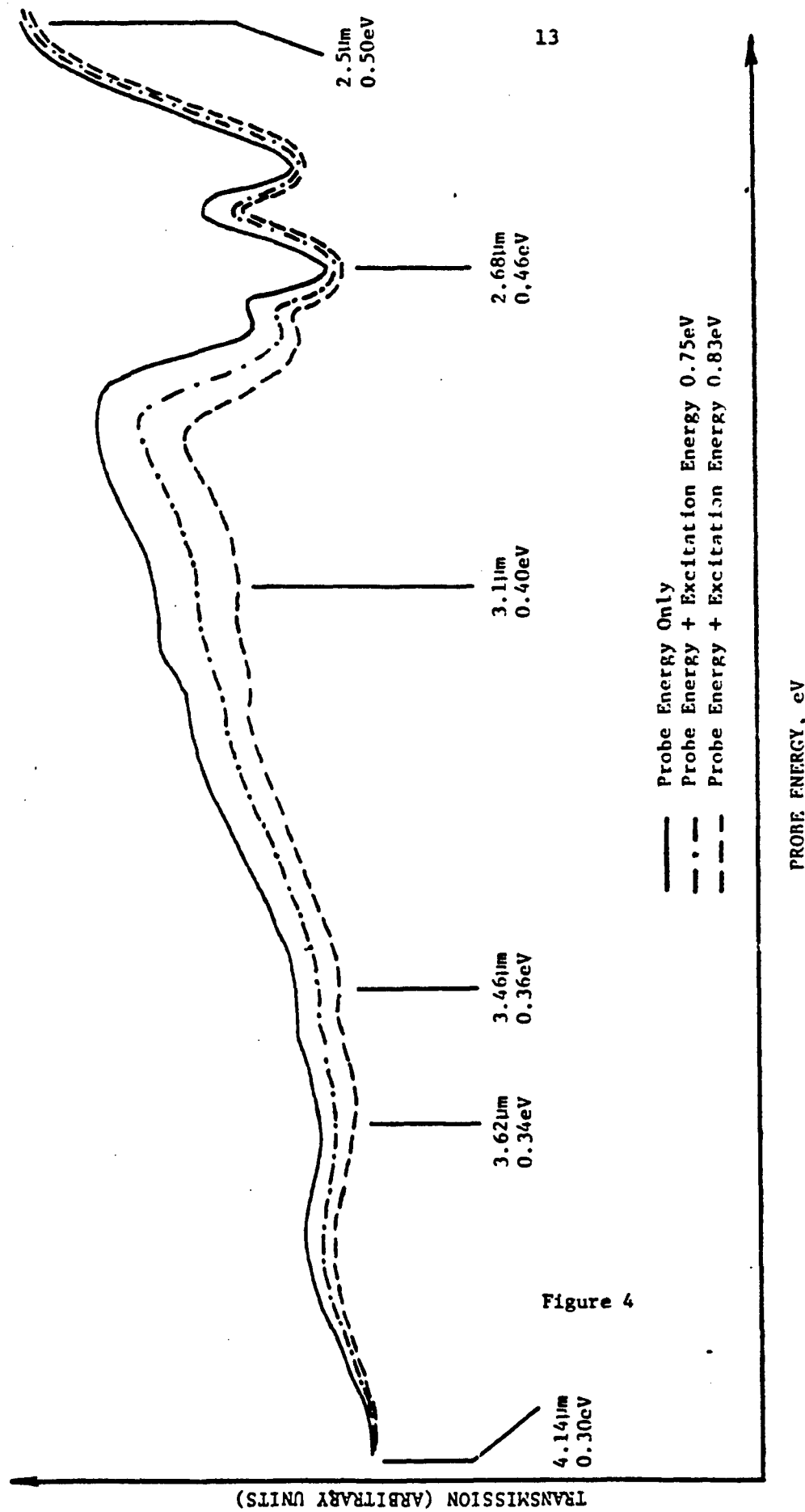
B. Conclusions; Divacancy Bands

The fact that the 3.62 μ m and 3.46 μ m bands are weak follows the results obtained by Rolfe⁶ where it is indicated that the bands are fluence dependent and the absorption coefficient is reduced by half at a fluence of 5×10^{18} neutrons/cm², see Figure 5.

The near edge absorption of 0.6eV also follows the results of Rolfe where it is shown that the effects of radiation cause band gap closure, see Figure 6. At room temperature (300°K) a band gap of ~0.51eV was observed in this work which is in close agreement with the results given in Figure 6 obtained by Rolfe.

The excitation energy of 0.81 eV - 0.89eV at first seems to be in contradiction with the knowledge of a band gap reduced to 0.6eV at 78°K. Based upon results from experimentation in the area of neutron radiation damage in silicon; however, a model is proposed here which explains these results with a logical interpretation based on the experimental facts.

First it is recognized that silicon is an indirect semiconductor in that the optical transition from valence to conduction



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11.

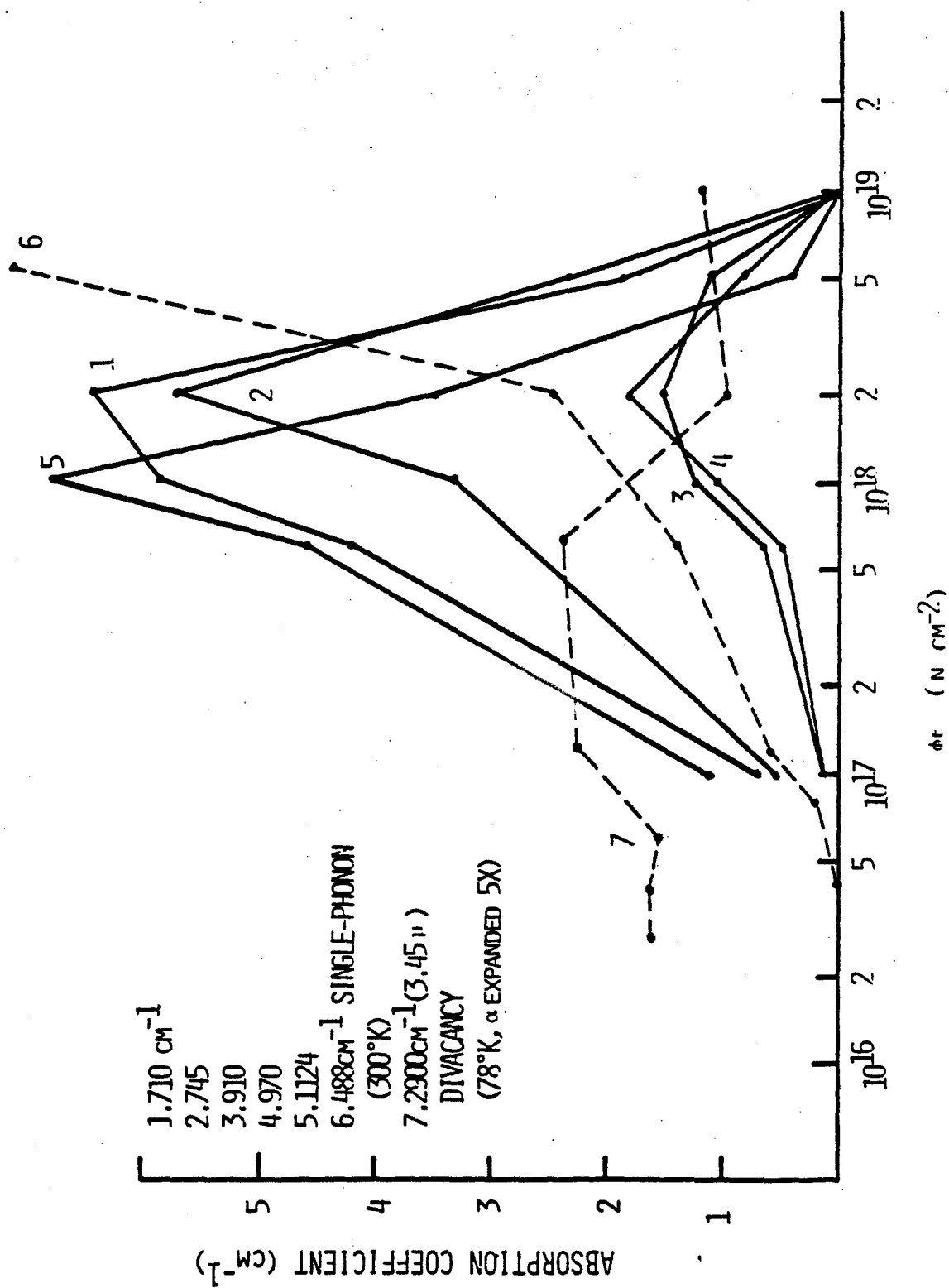


Figure 5

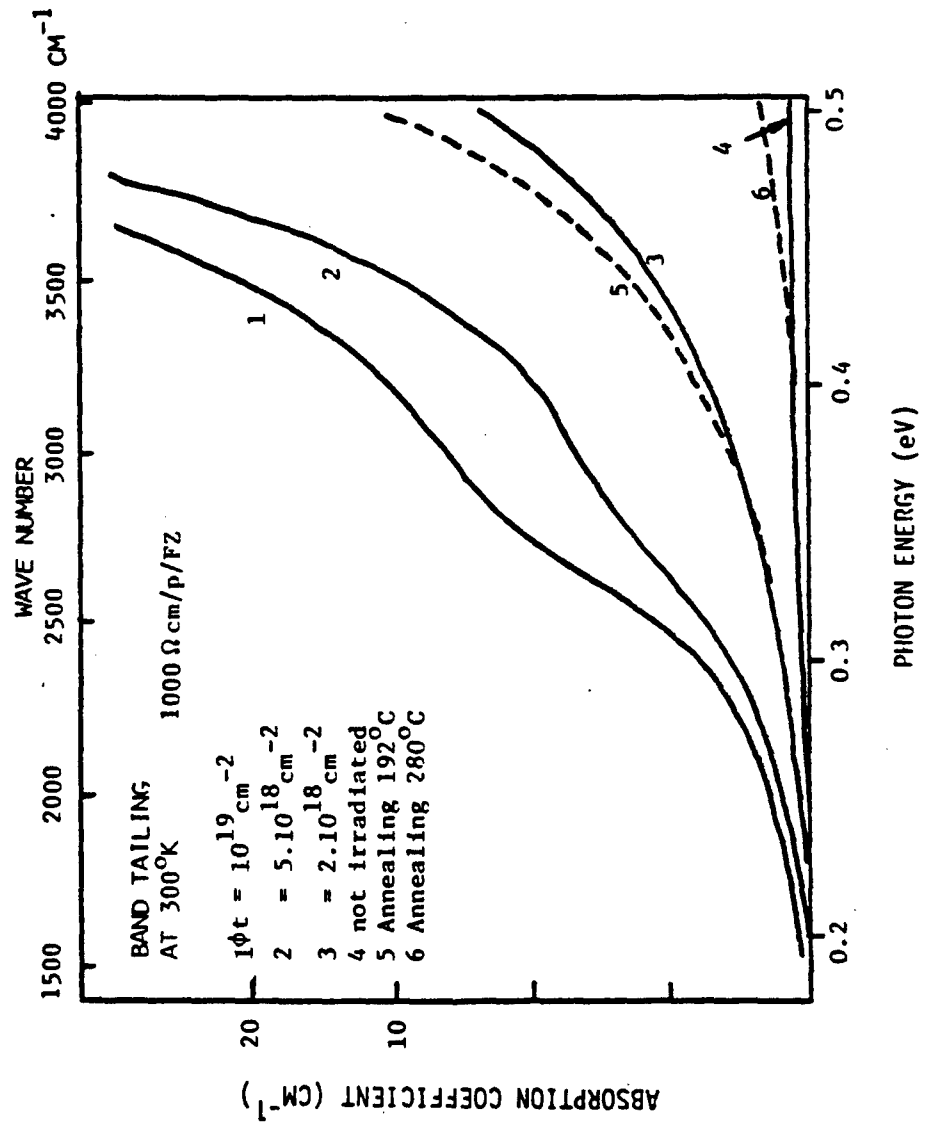


Figure 6

band is phonon assisted due to the maxima and minima of the two bands not lying at the same point in momentum space. After irradiation by high doses, such as, 5×10^{18} neutrons/cm² it has been shown⁶ that a large amount of disorder is produced in the lattice crystal structure and the band gap is narrowed. Since the indirect gap narrows, it is reasonable to assume that the direct gap narrows as well due to lattice disorder. If the direct gap narrows sufficiently enough, it is possible that the cross section for direct gap transition could be higher than that for the indirect transition in which the latter necessitates phonon assistance. Such a situation is proposed here and a model incorporating direct gap transition has been formulated.

Figure 7a depicts the model in the following manner. The direct gap has been reduced to 0.81eV thus causing the predominant transition from valence band to conduction band to be direct. Excitation energy (0.81eV) promotes an electron from the valence band to the conduction band by direct transition. The electron is captured by the positive divacancy charge state (V_2^+) which is photoionized to the neutral charge state (V_2^0). With the electron now in the V_2^0 state the probe light at 0.4eV promotes an electron from V_2^0 to the conduction band.

The V_2^+ charge state is the appropriate charge state to be photoionized for the following reasons. First, the predominant charge state will be dependent upon the Fermi level. The high fast neutron fluence to which the sample has been subjected here drives the Fermi level deep into the forbidden gap thus necessitating a V_2^+ or at the

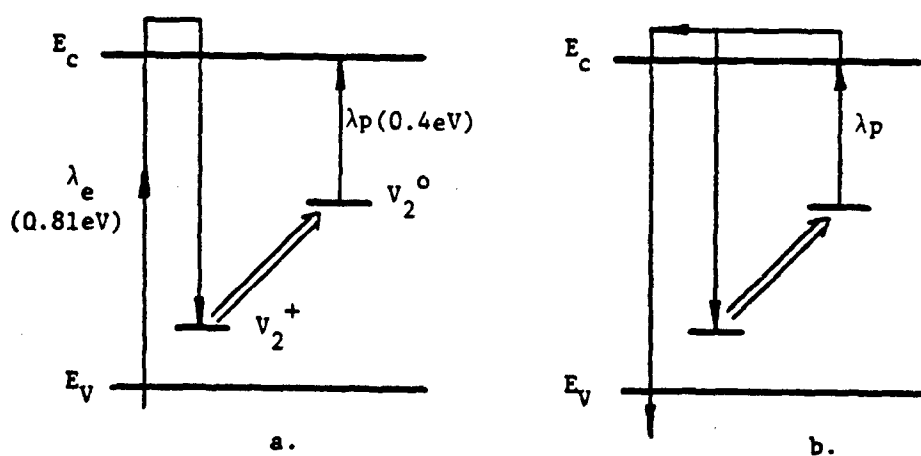
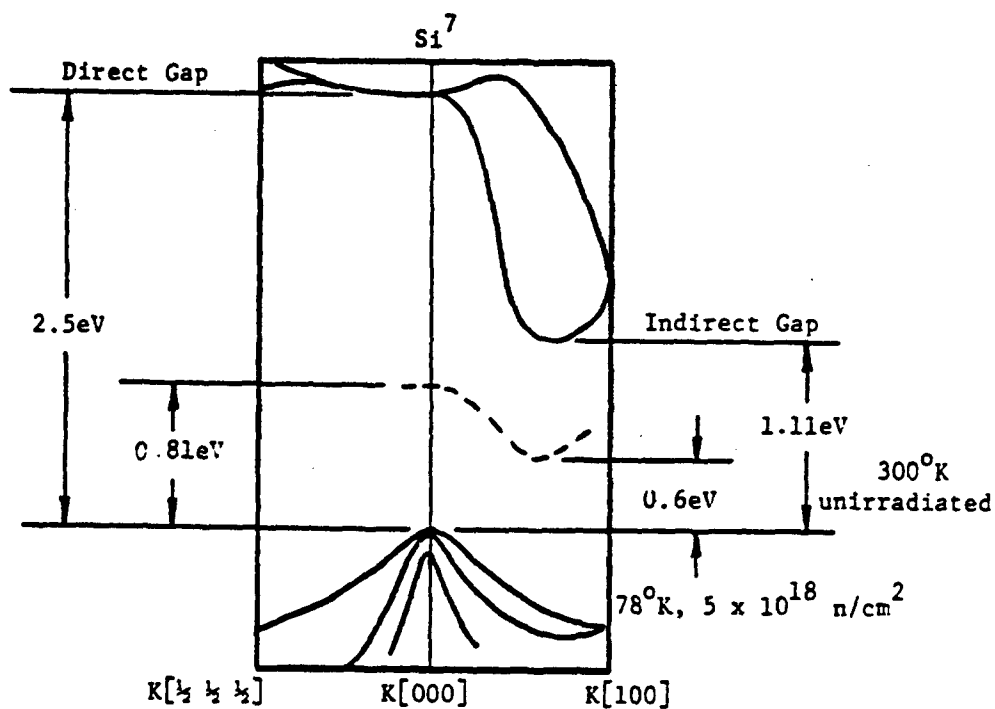


Figure 7

very least a V_2^0 charge state. Second, the fact that the 3.62 μ m and 3.46 μ m bands are observed indicate, as Carton-Merlet^{8,9} et. al. suggest, that a non-uniform Fermi-level due to the radiation damage does exist thus causing some V_2^- charge states to be present in the bulk sample. More importantly, however, the absorption bands are of low intensity thus indicating a small number of V_2^- charge states, and the intensity of the bands is unaffected by excitation light, see Figure 4: therefore, indicating the V_2^- charge state plays no part in the photoionization process. Thus, the photoionization process is $e(\text{C.B.}) + V_2^+ \rightarrow V_2^0$ and not $e(\text{C.B.}) + V_2^0 \rightarrow V_2^-$.

The cross section for direct transition vs. indirect transition is reflected in Figure 3 where it is shown that some absorption into the conduction band (0.6eV) by indirect transition and subsequent photoionization of the V_2^+ charge state does take place; however, it is significantly smaller due to lowering of the band gap causing pre-dominant absorption in the direct transition mode (0.81eV).

Figure 7b. reflects the light off decay sequence. The long absorption and decay times in Figure 2, T_2 and T_4 respectively, are indicative of the amount of trapping occurring at the V_2^0 charge state. This trapping along with the process of continuing photoionization of the V_2^+ charge state as the probe light promotes electrons from V_2^0 to the conduction band and then their subsequent capture by V_2^+ adds competition for electrons and a slower response for decay as compared to the absorption process.

The fact that the $3.1\mu\text{m}$ (0.4eV) absorption in Figure 4 is broad and not sharp is attributed to a defect energy level width which will be addressed later in this paper, or it can also be indicative of combinations of direct transitions and phonon assisted indirect transitions to various energy levels in the conduction band.

The width of the direct band gap excitation energy (0.81eV - 0.89eV) is due to the increase in optical absorption as higher energies are reached above 0.89eV.

IV

HIGHER ORDER BANDS

A. Experimental Results

The samples used were irradiated to 5×10^{18} neutrons/cm² ($E > 1$ MeV) with an initial resistivity of 200 Ω -cm p-type crucible grown and 1000 Ω -cm p-type float zone silicon. The samples were annealed to 500°C for 15 minutes. Two infrared absorption bands were investigated, 9.54 μ m (1048 cm⁻¹) and 9.08 μ m (1101 cm⁻¹) and these are in the spectrum shown in Figure 8.⁹ Probe light alone produced none of the higher order bands (HOB). A maximum absorption (intensity) for the 9.54 μ m and 9.08 μ m bands occurred for an excitation energy of approximately 1.17eV or band gap energy.

The absorption response of the probe light to the excitation energy for initial excitation is shown in Figure 9a. After the excitation light was turned off and a decay equilibrium reached, the sample was excited again with the same excitation light. After each excitation the absorption response would become faster until the absorption response became similar to the decay response when light was turned off (Figure 9b.). Initial signal level (prior to any excitation) was never achieved after the sample was exposed to excitation radiation. After subsequent excitations, the same equilibrium signal level was reached in the light off condition. Times for absorption and decay for both bands for initial excitation and subsequent excitations are shown in Table 1. Although both bands achieve maximum absorption with

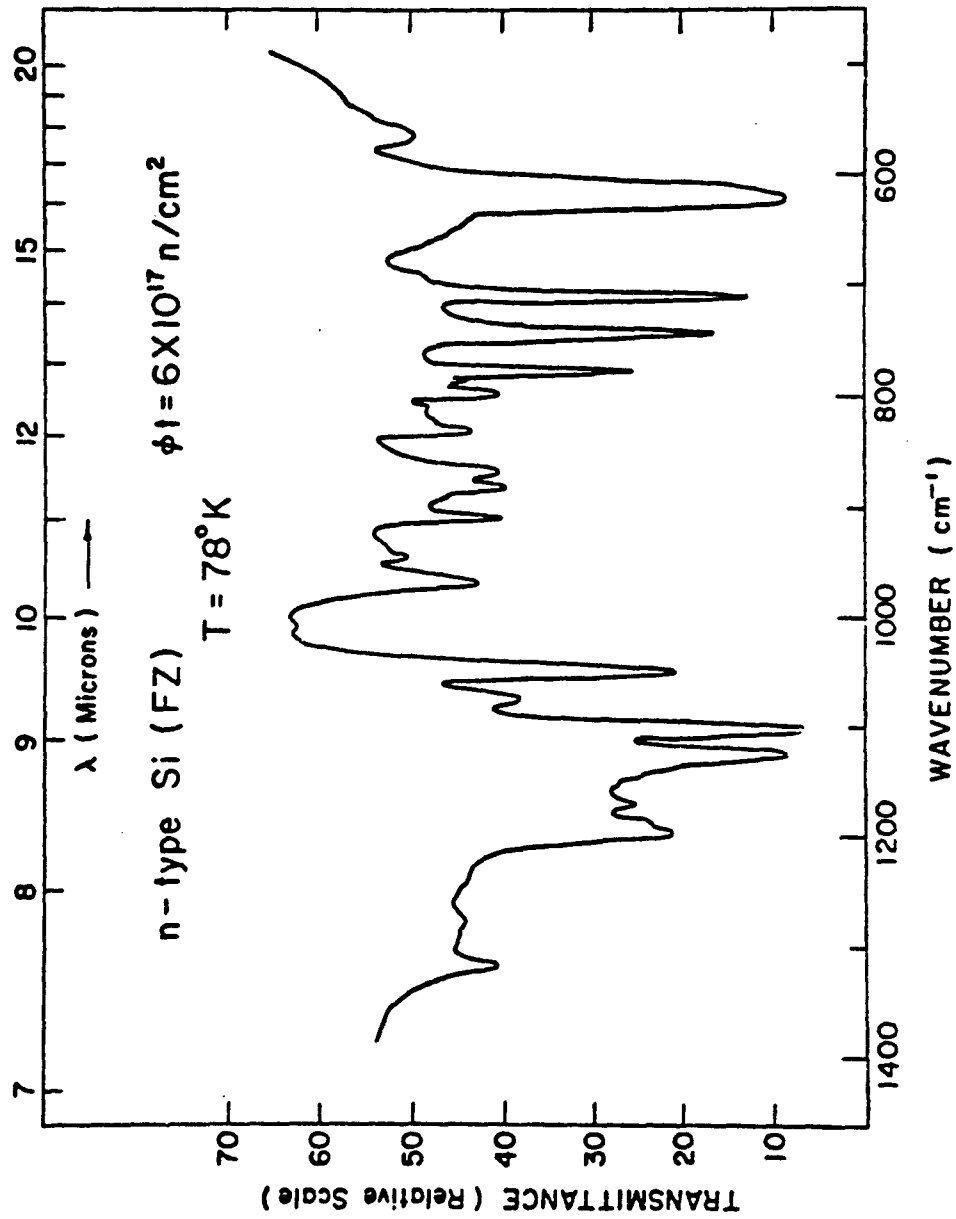


Figure 8

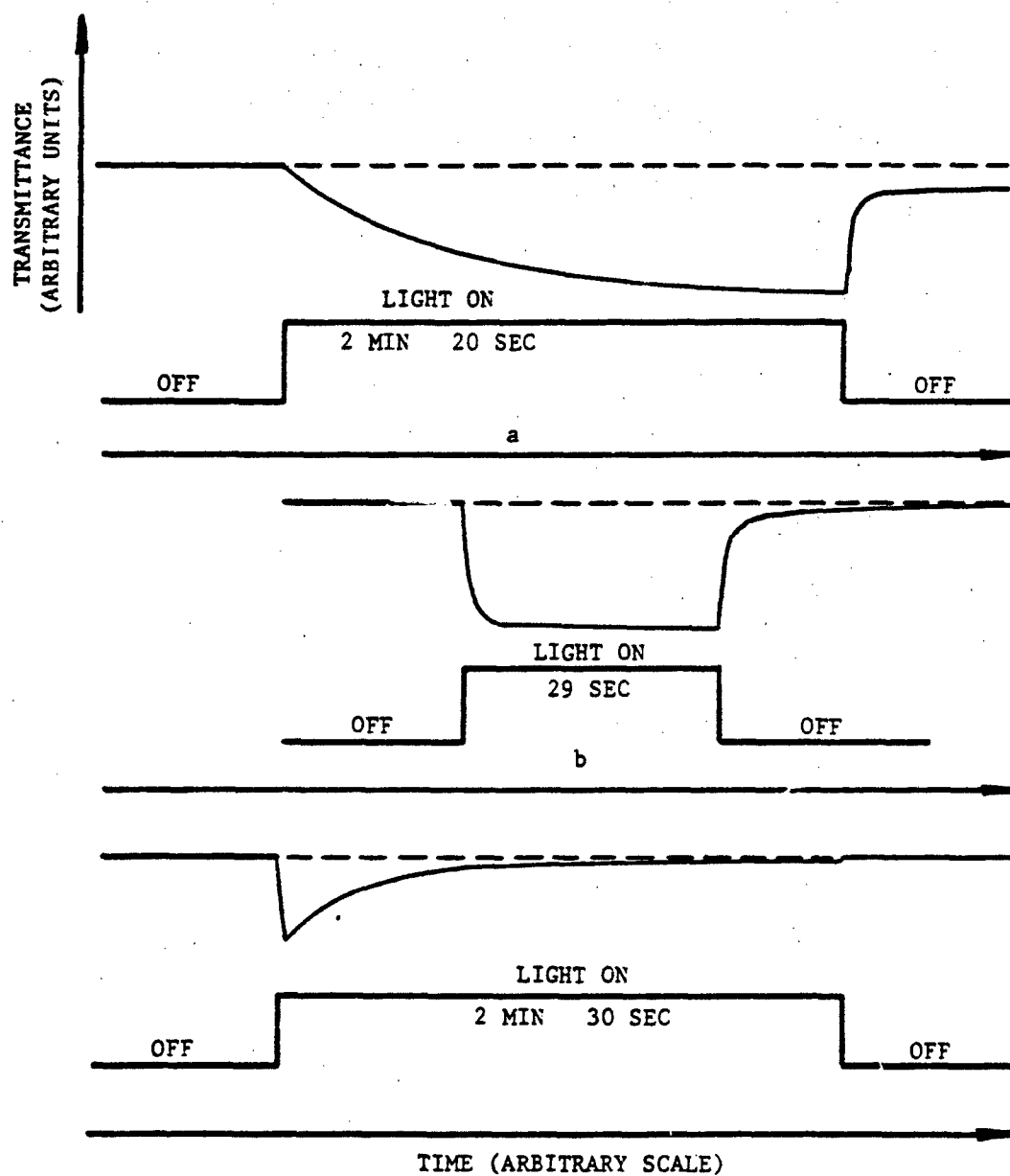


Figure 9

TABLE I

BAND	ABSORPTION TIME TO STEADY STATE	DECAY TIME TO STEADY STATE	EXCITATION	DEPLETION TIME TO STEADY STATE
9.54 μ m	2 min 20 sec	25 sec	INITIAL	
9.54 μ m	29 sec	31 sec	SURSEQUENT	
9.08 μ m	3 min 30 sec	1 min	INITIAL	
9.08 μ m	30 sec	20 sec	SURSEQUENT	
9.54 μ m			DEPLETION	2 min 30 sec
9.08 μ m			DEPLETION	4 min

the same excitation energy, the times for absorption and decay are different.

Subsequent excitation with the band gap energy was found to produce the initial response (Figure 9a.) only after waiting a considerable amount of time or after first exciting the sample with a lower energy. The most effective energy (hereafter referred to as the depletion energy) was found to be 0.74eV. The times for depletion energy equilibrium are shown in Table 1 and the depletion energy response is depicted in Figure 9c.

An interesting observation in experimental technique was made concerning the light beams. It was noticed that unless the excitation light beam was accurately superimposed on the probe beam illuminating the sample, the energy position for maximum absorption would change and shift upwards (i.e., 1.17eV to 1.25eV). This may be due to carrier diffusion where the higher energy electrons have a greater probability of diffusing to the probe beam and causing the absorption process. This effect of probe and excitation light position dependence in the sample was not observed for the excitation energy for the divacancy bands where, if the excitation light did not strike the sample at the same position as the probe light, the maximum absorption would occur at the same excitation energy but with less absorption.

B. Conclusions; Higher Order Bands

A model which this author believes correctly explains what is taking place with the excitation energy, probe beam, and depletion energy for both higher order bands is shown in Figure 10a. & 10b.

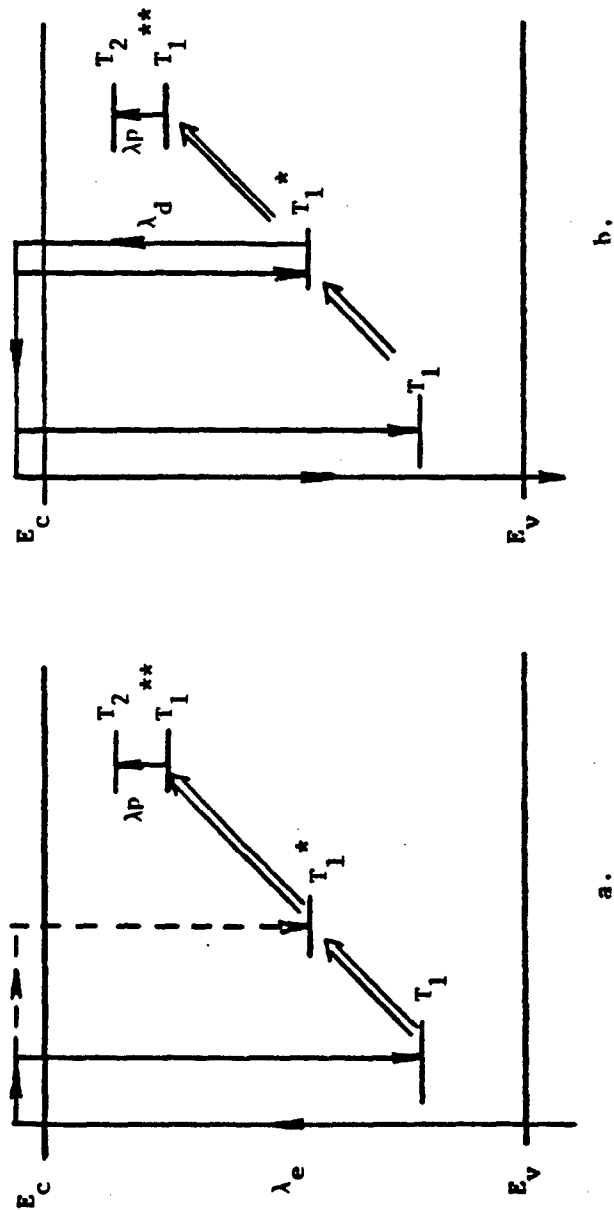
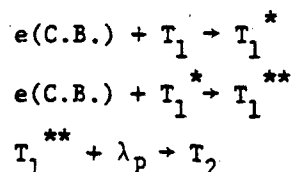


Figure 10

With initial excitation, electrons are raised into the conduction band from the valence band and then captured by a defect level of charge state T_1 . Upon capture of an electron T_1 changes to charge state T_1^* . This charge state also has an affinity for capturing electrons from the conduction band, and when this occurs, T_1^* changes to a new charge state T_1^{**} . Once this has been accomplished, the probe light (λ_p) excites an electron from T_1^{**} to a new defect level, T_2 , or an excited state of T_1^{**} . The process, therefore, is initially (see Figure 10a.):



When the excitation light is turned off, no more electrons are promoted into T_1 via the conduction band and those that were promoted have changed T_1 to T_1^* , also no more electrons are falling into T_1^* so that no more T_1^{**} states are being produced and those that were produced are almost totally eliminated by the continuing probe light. Therefore, what remains are the charge state T_1 and a quasi-stable charge state T_1^* . When the excitation energy is turned on again (subsequent excitation) the response is faster because T_1^* now exists where before it did not; so one observes $T_1^* + e(\text{C.B.}) \rightarrow T_1^{**}$, $T_1^{**} + \lambda_p \rightarrow T_2$ in addition to the initial response scheme.

The response to the depletion energy is depicted in Figure 10b. T_1^* is depleted as the depletion energy excites electrons from T_1^* to the conduction band. As this occurs, several competing processes

take place. First, some of the electrons initially removed from T_1^* to the conduction band fall back to T_1^* and are captured by some of the T_1^* charge states not yet depleted. This causes T_1^{**} to be produced and the initial absorption seen in Figure 9c. as the probe light makes the transition from T_1^{**} to T_2 . Electrons also fall into T_1 and initiate the absorption scheme previously observed for initial excitation. The depletion energy, however, will gradually deplete almost, if not all, the T_1^* charge states eventually as electrons are gradually lost to the valence band. Subsequent excitation with band gap light will then produce an excitation scheme and slow response almost exactly like that shown in Figure 9a., since the number of T_1^* charge states will be very low thus minimizing the quick response of capture of an electron by T_1^* immediately and forming T_1^{**} . The fact that an exact initial response is not reproduced indicates that some (although relatively small number) T_1^* states may exist, further indicating some level of trapping at T_1^* . There may be some trapping at T_1^{**} as well, and this in addition to electrons possibly being lost to other defects may be contributing factors as to why initial equilibrium level is not reached.

All three charge states are necessary to explain the absorption, decay and depletion energy responses. If there was just one charge state T_1 with an excited state or new defect level T_2 , then each subsequent response to excitation would be the same and would be relatively fast since there would be no competition and other charge states would not be produced. In addition no depletion energy would

have been realized since T_1 would have been eliminated by the probe light, which is not the case. There is a depletion energy and the initial excitation response is slow and subsequent excitations are faster. If there were just two charge states T_1 and T_1^{**} with a defect level T_2 , the response again would be expected to be the same on subsequent excitations, since any T_1^* would be depleted by the probe light to T_2 and any electron captured by T_1 would have caused a charge state change to T_1^* . The combination of observed responses to initial excitation, subsequent excitation and depletion energy, therefore, dictate the necessity for three charge states and the above model.

This model and its experimental findings are not in disagreement with the findings of Koval et. al.¹¹ Their results indicate through use of filters that the HOB can be separated into two groups (Corelli et. al. indicate more groups through stress analysis).⁴ Within one of the groups the 9.54 μ m and 9.08 μ m bands can be observed with all wavelength light and also with a 2 μ m long pass filter. In the latter case energy from the valence band to T_1 and T_1^* would have been passed thus causing the same absorption responses.

Investigation of the valence band to T_1^* transition failed to produce any results; however, this was almost expected since the T_1^* charge state is dependent upon the photoionization of the T_1 charge state and the excitation energy used in searching for the valence band to T_1^* charge state would not have matched the photoionization energy of T_1 from the valence band. Search for the valence band to the T_1 charge state transition showed one absorption utilizing probe light

alone at $\lambda_p = 7.3\mu\text{m}$ (0.17eV); however, due to the low intensity and the fact that the $7.3\mu\text{m}$ absorption band did not go out with the other HOB after annealing to 606°C , this energy could not be confirmed as the T_1 charge state level.

In addition to the $9.54\mu\text{m}$ and $9.08\mu\text{m}$ bands, band gap excitation energy also produced in varying intensities the $8.4\mu\text{m}$ (1189cm^{-1}), $10.3\mu\text{m}$ (970cm^{-1}), $11.0\mu\text{m}$ (910cm^{-1}), $11.5\mu\text{m}$ (868cm^{-1}), $12.9\mu\text{m}$ (776cm^{-1}), and $13.5\mu\text{m}$ (741cm^{-1}) bands. Other bands were not noted; however, this could be due to the fluence dependence of the HOB. These bands* were also not completely checked with the aforementioned model; therefore, it is not known whether they follow the same pattern as the $9.54\mu\text{m}$ and $9.08\mu\text{m}$ bands for absorption and depletion.

* The time required to complete the studying of all HOB bands is sufficiently long as to go beyond the scope of the study undertaken in this thesis. Additional studies are needed to complete the task of using the technique reported herein to make detailed studies of all bands.

V

SUMMARY

As the experimental results indicate, this new optical system offers the ability to observe the time-energy dependence for population of an excitable state through electron infrared absorption and the subsequent depopulation once the excitation light pulse is turned off. New properties of defects, such as, more defined energy levels, photo excitation cross sections, excitation and decay times of energy levels and better understanding of charge states associated with particular bands can be obtained from this experimental technique.

One new characteristic currently being thought of is the possibility of varying widths to these defect energy levels and charge states. The width of excitation energies, depletion energies and regions of absorption response seem to indicate such an energy width. Past experiments in photoconductivity¹² also suggest such a characteristic. Figure 11¹² shows that the photoconductivity is not instantaneous once a particular energy is reached but varies over a width of energy. In one case the width is 0.2-0.65eV, whereas, in another it is relatively sharp, 0.55-0.6eV.

A new experiment incorporating the optical method reported here and photoconductivity is being contemplated for future research. Such an experiment with variations in sample temperature, irradiation, fluence, initial resistivity and oxygen content should bring about new and definitive results on infrared absorption bands, their associated charge states, and other characteristics.

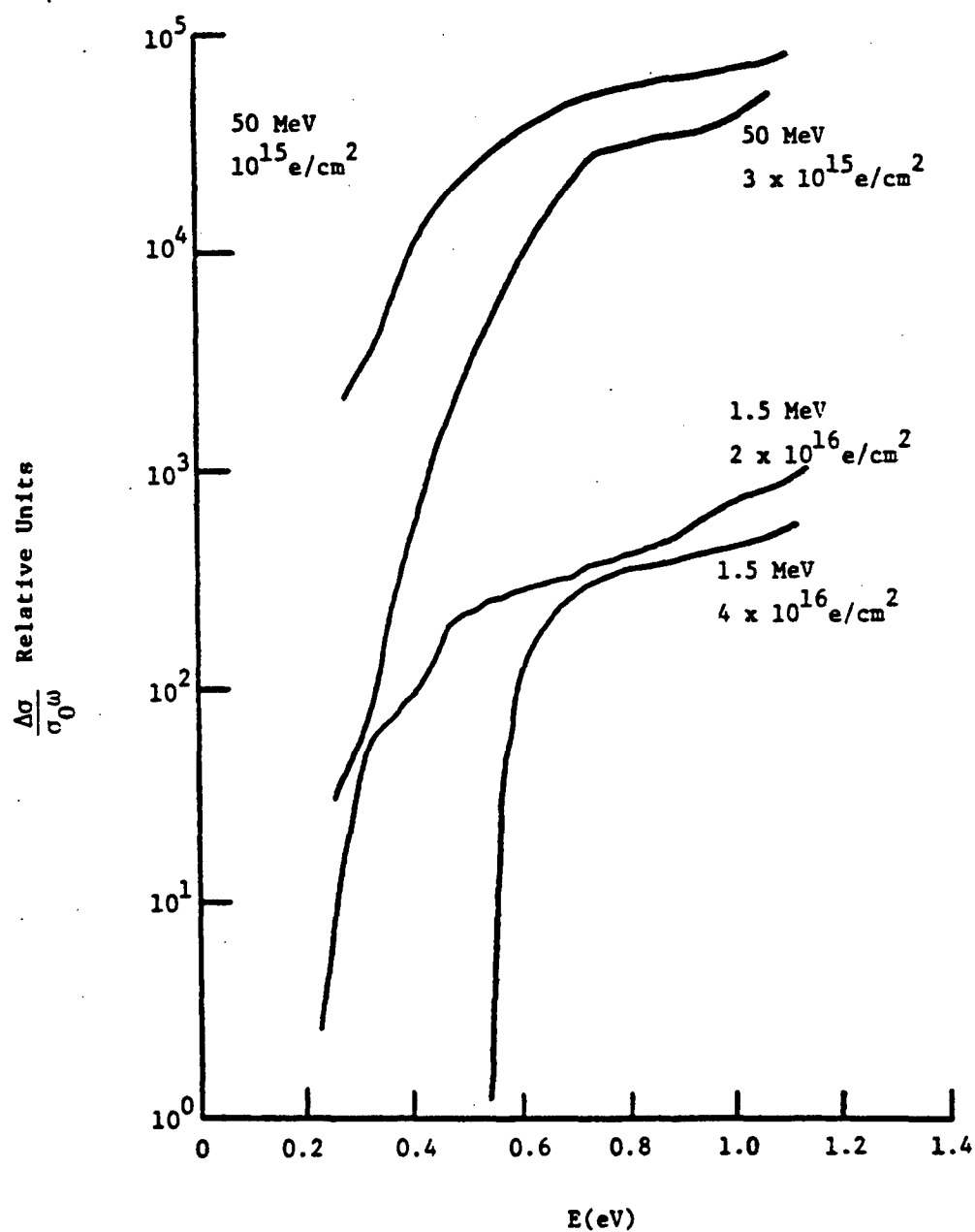


Figure 11

An important and concluding result of the optical technique developed by this research is that it can be used to investigate defects and their associated infrared absorption bands whenever one is dealing with defect levels for which it is possible to promote electrons to or out of the levels.

VI

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> A new optical method incorporating two monochromatic light beams incident on the sample whose temperature is at a 78°K is used to study infrared active radiation-induced defect absorption bands. Defect energy levels, photoionization excitation and deexcitation processes were examined for the divacancy associated bands found in the 3 - 3.6 μ m wavelength region and two higher order bands which appear in the 7 to 14 μ m wavelength region.

For the divacancy band region 10 μ m-cm and 0.10-cm p-type (P-doped) crucible grown silicon irradiated to 5×10^{18} neutrons/cm² (E>1 MeV) were examined. An excitation energy (E_c) of width 0.81 eV to 0.89eV was found to produce an absorption throughout the entire 3.3 μ m absorption band region with a maximum absorption occurring around 3.1 μ m. A model is proposed which incorporates direct band gap transitions.

A 200 μ m-p-type (B-doped) crucible grown silicon sample irradiated to 5×10^{18} neutrons/cm² (E>1 MeV) and annealed at 500°C for 15 minutes was examined for higher order bands. The 9.54 μ m and 9.08 μ m bands were found to have a maximum absorption for band gap energy. A model is proposed for the photoionization process incorporating the band gap energy for absorption and a depletion energy of 0.74eV. ←